

July 6, 2004

DECLARATION

The undersigned, Dana Scruggs, having an office at 8902B Otis Avenue, Suite 204B, Indianapolis, Indiana 46216, hereby states that she is well acquainted with both the English and German languages and that the attached is a true translation to the best of her knowledge and ability of PCT/EP 03/00827 (INV.: MOEHL, W., ET AL), entitled "Method for Coating the Quartz Burner of a HID Lamp".

The undersigned further declares that the above statement is true; and further, that this statement was made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or document or any patent resulting therefrom.

A handwritten signature in black ink, reading "Dana Scruggs", with a long, sweeping horizontal stroke extending to the right.

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Dana Scruggs

*1/20/04*

1 METHOD FOR COATING THE QUARTZ BURNER OF A HID LAMP

2

3 The present invention relates to a method for coating a quartz burner of a HID  
4 lamp with a UV-reflecting layer system.

5

6 For illumination purposes with high illuminance, high-intensity or high intensity-  
7 discharge (HID) lamps are used which have a plasma quartz burner as the lamp,  
8 the lamp being located inside a tubular jacket.

9

10 UV photons, which excite the atoms in the plasma to generate light in the visible  
11 range, play a central role in generating light in the plasma in the burner of HID  
12 lamps. With the known HID lamps, however, large UV photon fluxes leave the  
13 quartz burner unused. As such, the energy efficiency of the HID lamp is reduced.

14

15 The unused escaping of the UV photon fluxes could be reduced significantly if  
16 the quartz burner can be provided with a UV-reflecting layer system, which must  
17 be able to last for the entire service life of the lamp, however. Due to the  
18 aggressive plasma medium in the quartz burner and the high operating  
19 temperatures of the burner, this cannot be easily ensured, however.

20

21 UV-reflecting layer systems on different substrates are known from various  
22 publications, and they will be described individually hereinbelow. These known  
23 layer systems have not resulted in the desired success with quartz burners for  
24 HID lamps. One reason is that the coating step itself has proven difficult. Another  
25 reason is that no notable improvements in the energy balance were achieved.

26

27 The object of the present invention is to create a method for coating a quartz  
28 burner of a HID lamp with a UV-reflecting layer system which is capable of being  
29 applied to the quartz burner with relatively simple means, which has a sufficient,  
30 adapted service life, and brings about a notable improvement in the energy  
31 balance.

1 This object is attained according to the present invention by alternately applying  
2 amorphous thin layers made at least of titanium oxide and silicon oxide having  
3 the general stoichiometry  $\text{TiO}_y$  and  $\text{SiO}_x$  by means of a PICVD method at high  
4 power density and increased substrate temperatures ranging from  $100^\circ$  to  $400^\circ$   
5 C, using small growth rates ranging from 1 nm/sec to 100 nm/sec so as to form  
6 an interference layer system having a thickness of less than 1200 nm and a  
7 minimized UV-active defective spot rate.

8  
9 The design, according to the invention, of the interference layer for the UV-  
10 reflecting layer system composed of amorphous titanium oxide and silicon oxide  
11 layers, the standard elements, using small growth rates at temperatures ranging  
12 from  $100^\circ$  to  $400^\circ$  C ensures, for the service life of the HID lamp, that UV photons  
13 which would otherwise escape from the quartz burner can be recaptured to a  
14 significant extent via reflectance in the plasma in the quartz burner for further  
15 generation of light in the visible range, thereby significantly improving the energy  
16 balance of the HID lamp. It has been shown that, at a wavelength of 360 nm, the  
17 interference coating according to the present invention results in a UV  
18 reflectance of 70%.

19  
20 The PICVD method used, which is known sufficiently per se and therefore need  
21 not be explained in greater detail here, makes it possible – due to particular  
22 process parameters – to deposit the interference coating in the interior of the  
23 quartz burner and on the quartz burner in a relatively simple manner.

24  
25 With regard for an inner coating, it has also been shown, surprisingly, that the  
26 inner coating is inert in the presence of the aggressive plasma medium.

27  
28 The coating according to the present invention suffices, surprisingly, with the  
29 standard elements Si/Ti, without any doping elements such as C, H, N, and  
30 without heteroelements such as Al, Nb, Ta.

31

1 The use of the PICVD method also enables application on corresponding  
2 multistation systems, which advantageously results in a high throughput rate in  
3 the coating of quartz burners, in particular.

4  
5 The interference coating has a thickness of  $< 1200$  nm, preferably  $< 500$  nm.  
6 Layers of this type are highly flexible. Furthermore, intrinsic stresses—which  
7 could cause the layer to peel off—can also be prevented with thin layers of this  
8 type. A typical layer construction includes approximately 50 alternating individual  
9 layers of Ti/Si oxide, with layer thicknesses of between 5 nm and 100 nm,  
10 whereby the thicknesses need not necessarily be distributed strictly equally.  
11 Instead, accumulations of small layer thicknesses can also occur; this depends  
12 on the design. The distribution of the layer thickness in the layer packet is  
13 therefore a mix in which thicknesses of 5 nm to 100 nm can occur in a mixed  
14 manner, e.g., extremely thin layers can occur frequently.

15  
16 The very low attainable defective spot rate ensures very low UV absorption in the  
17 interference coating. What is meant here are structural defective spots, i.e., a  
18 very low inclusion of foreign elements. To improve the understanding of the  
19 present invention, the following will be noted:

20  
21 In a quartz burner for HID lamps, a plasma is ignited in an appropriate glass  
22 jacket—the discharge vessel—in a gas mixture composed of metal halogenides  
23 and starter gas, e.g., xenon, with DC/AC at 100 to 300 Hz. UV photons bring  
24 about electron transitions in the atomic shells of the gases and emit:

- 25
- 26 a. Visible light, in a portion of approximately 40%; this yield should be as  
27 high as possible.
  - 28
  - 29 b. UV light. This portion is returned by the coating according to the  
30 present invention, and the additional photons obtained as a result bring

about a higher yield of light waves in the visible range. As a result, the same light yield is possible while saving current.

c. An infrared portion, which can also be reflected.

Due to these effects, the burner surface with the reflectance layer packet according to the invention becomes, practically speaking, a band pass that allows only the light waves in the visible range to pass and reflects the wavelengths in the UV range or in the UV and infrared range into the interior of the burner.

Due to its UV-absorbing property,  $\text{TiO}_2$  is normally unsuitable for use as a UV reflector.  $\text{SiO}_2$  is reflecting, however, and has no absorption losses. Contrary to expectations,  $\text{TiO}_2$  with  $\text{SiO}_2$  as the usable reflecting layer is capable of being used in the layer design with 70% efficiency at  $\lambda = 360 \text{ nm}$ . The important point here is the defective spot rate, in order to attain the lowest absorption possible. The layer materials are deposited amorphously in the oxidic form at  $350^\circ \text{C}$ .

As a result of the measure according to the present invention, it is possible, contrary to expectations, to obtain reflecting interference systems inside and/or outside having high UV efficiency (absence of defective spots) and inertness in the presence of the plasma medium in the quartz burner for quartz burners for HID lamps by using types of alternating layers of  $\text{TiO}_2/\text{SiO}_2$  known per se using small growth rates and high, preferably constant deposition temperatures which result in an improvement in the energy balance of these types of lamps.

Publication DE 199 62 144 A1 shows a UV-reflecting interference layer system composed of alternating layers of titanium oxide/silicon oxide for transparent substrates such as filters, eyeglasses, and for tubular jackets of discharge lamps, to increase the emitted light in a color-neutral manner while simultaneously

1 increasing the UV protective effect. The publication also mentions deposition  
2 using chemical vapor deposition, supported with plasma, in particular.

3  
4 In the case of the present invention, it is not the tubular jacket of the HID lamp  
5 that is coated, but rather the burner itself that is located therein, whereby,  
6 contrary to expectations, the layers applied to the burner according to the  
7 invention are inert in the presence of the aggressive plasma medium, which  
8 makes internal coating of the burner possible. Nor does this publication disclose  
9 the plasma impulse chemical vapor deposition method, i.e., the PICVD method,  
10 or, concretely, the process parameters used according to the invention.

11  
12 The paper authored by M. Walther, et al., entitled "Multilayer barrier coating  
13 system produced by plasma-impuls chemical vapor deposition (PICVD)" in  
14 "Surface and Coatings Technology 80 (1996) 200 – 202", and DE 44 38 359 C2  
15 describe methods for applying barrier layers composed of alternating layers of  
16  $\text{TiO}_2/\text{SiO}_2$  on plastics at relatively low temperatures. In the case of the present  
17 invention, deposition is carried out at higher temperatures (e.g.,  $350^\circ\text{C}$ ) and in  
18 combination with small growth rates. This is the only way to create a UV-suitable  
19 absence of defective spots and inertness in the presence of the plasma medium.  
20 The layers known from the aforementioned publications do not fulfill these  
21 criteria.

22  
23 Publication DE 195 30 797 A1 describes a composite material for protection  
24 against radiation in greenhouse systems, comprising a transparent carrier and an  
25 interference layer system deposited thereon, composed of alternating layers of  
26  $\text{SiO}_2/\text{TiO}_2$ , among other substances. In this known case, vapor deposition layers  
27 are described that are entirely unsuitable for applications on lamp burners, in  
28 particular on the inside, due to their column growth.

29  
30 Publication DE 44 32 315 A1 describes a mercury vapor lamp with a short arc  
31 having a discharge vessel made of quartz glass that is provided with a multilayer

1 interference reflectance filter made of  $\text{TiO}_2/\text{SiO}_2$  on the outside to suppress  
2 undesired, short-wave UV radiation below 365 nm, and which has an absorbing  
3 titanium oxide layer on the inside. In the case of the invention, however, the inner  
4 layer is also designed to be reflective. Furthermore, the deposition of layers using  
5 the PICVD method with the process parameters according to the invention is not  
6 described in the aforementioned publication, i.e., the known layers would be  
7 unsuitable for coating the burner of a HID lamp.

8  
9 Publication DE 41 15 437 A1 shows a projection cathode ray tube with an optical  
10 multilayer interference filter for increasing the emitted light stream. This known  
11 layer system would also be entirely unsuitable for coating the burner of a HID  
12 lamp, because it is not deposited using the measures according to the invention.

13  
14 Finally, publication DE-PS 34 30 727 shows an incandescent lamp with a sealed  
15 glass bulb and an optical interference film on the inner and/or outer surface of the  
16 glass bulb. The known layer system is only IR-reflecting, however; there is no  
17 mention of UV-suitability. Furthermore, the known layer system would not be  
18 suitable for coating the burner of a HID lamp, either, because it is not applied to  
19 the substrate using the measures according to the invention.

20  
21 Preferably, a method for coating is provided, via which the titanium oxide and  
22 silicon oxide layers having the stoichiometry  $\text{TiO}_2$  and  $\text{SiO}_2$  are deposited by  
23 controlling the deposition parameters of the PICVD process. It has been shown  
24 that the best reflectance results are obtained by using alternating layers in  
25 accordance with the aforementioned "ideal stoichiometry".

26  
27 A particularly low-stress coating in terms of intrinsic stresses may be obtained  
28 using a method via which a layer system having a thickness of less than 500 nm  
29 is applied.

30

Particularly good deposition results are obtained with a PICVD method in which a pulsed microwave method having a fundamental frequency of 2.45 GHz is used to generate the plasma, and in which the substrate formed by the quartz burner is maintained at a constant deposition temperature.

The substrate temperature can be held constant in a simple manner using a process method in which an O<sub>2</sub> plasma is run for substrate heating and the temperature is monitored optically by measuring the substrate surface.

Since the reflectance quality of the layer system is determined in a decisive manner by the PICVD method, a method is provided according to a further development of the invention, via which the process parameters for the PICVD method for applying the alternating layers of TiO<sub>2</sub>/SiO<sub>2</sub> with a constant substrate temperature achieved using O<sub>2</sub>-plasma substrate heating are selected as follows:

Parameter	Substrate heating, O <sub>2</sub> plasma	Layers (TiO <sub>2</sub> /SiO <sub>2</sub> )
Process pressure (mbar)	0.2	0.1 – 0.5
Total mass flow (sccm)	100	100 – 500
Precursor concentration	-	0.1 – 5%
MW power (%)	70	30 – 60
Pulse duration (ms)	1 – 2	0.1 - 2.5
Pulse pause (ms)	2 – 4	10 – 300
Constant temperature (° C)	350	350

Since the layer systems according to the invention are surprisingly inert in the presence of the plasma in the quartz burner, it is advantageously possible to coat the quartz burner on the inside of its jacket to achieve a high rate of recapture of UV photons.



1 It is also possible, however, to coat the quartz burner on the outside of the jacket,  
2 either as an alternative or in addition to the inner coating.

3

4 Since the method is robust and stable, on-line control of the layer growth is not  
5 carried out. Constant deposition rates can be run, with the advantage that the  
6 measurement of the layer thickness is reduced to a process of counting the  
7 microwave pulses.

8

9 The invention will be described in greater detail with reference to an exemplary  
10 embodiment shown in the drawing.

11

12 Figure 1 is a schematic depiction of the design of a HID lamp with the coating  
13 according to the invention, shown as an enlargement of a section, and  
14

15 Figure 2 is a block diagram of the design of a modified PICVD device for  
16 applying the layers according to the invention.

17

18 HID lamp 1 depicted in Figure 1 is composed of a tubular jacket 2 and a quartz  
19 burner 3 with electrodes 4. "HID" is a technical term that stands for High Intensity  
20 Discharge.

21

22 The specific design and function of an HID lamp 1 of this type is known and  
23 therefore need not be explained further. As shown in the associated enlargement  
24 of a section, a UV-reflecting layer packet 5 is applied to the inner surface of  
25 burner wall 3a, the layer packet being composed of a large number, e.g., 50, of  
26 individual alternating layers made of Ti/Si oxide. Unshaded layers 5a represent Ti  
27 oxide layers, and shaded areas 5b represent Si oxide layers. The thickness of  
28 the individual layers typically ranges between 5 nm and 100 nm, whereby the  
29 thicknesses need not necessarily be distributed strictly equally; instead,  
30 accumulations of small layer thicknesses can also occur. This depends on the  
31 design. The layer thickness is preferably < 1200 nm, however, because the layer

1 then has high flexibility, and intrinsic stresses are prevented. As the number of  
2 individual layers increases, the layer thicknesses are therefore kept  
3 correspondingly smaller.

4

5 Layers 5a, 5b are amorphous Si/Ti oxide thin layers having the general  
6 stoichiometry  $\text{TiO}_2$  and  $\text{SiO}_2$ , because this allows the best reflectance results to  
7 be obtained.

8

9 Figure 2 shows a block diagram of the design of a device for the deposition of the  
10 layer system according to the invention on the inside of burner wall 3a. This is a  
11 PICVD system with a vacuum chamber 6 that accommodates quartz burner 3, in  
12 which a vacuum is maintained using a vacuum arrangement 7, and a pulsed  
13 plasma with a fundamental frequency of 2.45 GHz is generated using a  
14 microwave generator 8 coupled in at 8a.

15

16 The necessary gases are fed from a stage 9 into vacuum chamber 6, namely the  
17 gas in which the plasma is ignited—oxygen in this case—and, in an alternating  
18 manner, so is the particular precursor, out of which the Ti oxide and Si oxide  
19 layers are deposited in alternating fashion in combination with the oxygen  
20 plasma. The possible precursor gases for deposition of the aforementioned  
21 layers are sufficiently well-known. A process control 10 controls the entire course  
22 of the procedure, in particular the growth rate of the layers and their thickness.  
23 An on-line control of the layer growth is not necessary, because the process is  
24 robust and stable. Constant deposition rates can be achieved, so that the  
25 measurement of layer thickness is reduced to a procedure of counting microwave  
26 pulses. Another essential feature for the layer design is that deposition take place  
27 at increased substrate temperatures. In this case, quartz burner 3 is heated in  
28 simple fashion to approximately 350° C, for example, using the oxygen plasma,  
29 as is described in DE 40 08 400 C1 (column 8), for example. The layer materials  
30 of the precursor are then deposited amorphously in the oxidic form at 350° C. In  
31 general, the substrate temperature can be between 100° and 400° C.

1

2 Typical process parameters are:

3

Parameter	Substrate heating, O <sub>2</sub> plasma	Layers (TiO <sub>2</sub> /SiO <sub>2</sub> )
Process pressure (mbar)	0.2	0.1 – 0.5
Total mass flow (sccm)	100	100 – 500
Precursor concentration	-	0.1 – 5%
MW power (%)	70	30 – 60
Pulse duration (ms)	1 – 2	0.1 - 2.5
Pulse pause (ms)	2 – 4	10 – 300
Constant temperature (° C)	350	350